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RECOVERY OF HEAVY METALS FROM WASTE PLATING SOLUTIONS AT COMDEV

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RECOVERY OF HEAVY METALS

FROM WASTE PLATING SOLUTIONS AT COMDEV

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DISCLAIMER

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1.0 ABSTRACT

The following report details the operating results of a waste treatment system which was procured and was installed in order to treat major sources of chemical waste generated at COM DEV plating facility. This system emphasizes recovery of metallic waste through the use of electrolytic (EMR) cells. The objective of the waste treatment operation is to recover heavy metals including Zinc (Zn), Nickel (Ni), Copper (Cu) and Silver (Ag) from plating waste solutions. The system allows COM DEV to meet government standards of the Regional Municipality of Waterloo. It also minimizes waste sludge produced, space requirement, hazardous materials handling, plating bath contamination and liability. A series of waste treatment experiments have been performed at COM DEV and the results confirm that this waste treatment system operates effectively to reduce metal concentration to less than a half of the Municipal sewer use by law (government standard). A total cost saving with the EMR system exceeds $12,000 per year and the capital cost of the recovery portion of the system pays back within 4½ years.

2.0 INTRODUCTION

2.1 General Background

COM DEV was founded in 1971 and over the years began the process of building a world class high technology product line. Since COM DEV moved into a new 20,000 square foot building in Cambridge, the building as well as employee population has kept growing.

COM DEV's largest business area is that of satellite borne subsystems. The company is expanding the scope and nature of its products and capabilities and increasing its effort to sell them in a wider variety of markets.

As part of its in-house manufacturing capabilities, COM DEV has developed a space industry approved plating facility. Production processes center on zinc, nickel, copper and silver plating as well as smaller scale capability in gold and rhodium. The new waste treatment system is intended to handle current and future requirements for our plating facility as well as those of COM DEV as a whole.

The metal plating operation has been strongly effected by the recent trends relating to environmental issues. Discharge regulations have been strengthening steadily and are expected to continue to do so in the foreseeable future. As public concern regarding the environment increases, more restrictive regulations with higher fines are expected. These unavoidable trends, coupled with rising water and sewer rates, and limited waste disposal options, force the metal plating operation to material conservation and recovery. Pollution abatement technologies have, therefore, centred on the reduction, reuse and recovery.

One of the pollution sources in the metal plating operation is heavy metals such as COM DEV uses in salt form. Heavy metals, which play absolute importance in hardware manufacturing processes in the space industry, are used in acid or base baths. The plating operations, being complex and diverse, require large quantities of water for rinsing.

Requirements for COM DEV to control wastes in the metal plating operation, are such that all possibilities including financial resources, available space, future regulations and maintenance have been considered for the decision and operation of COM DEV's Electrolytic Metal Recovery (EMR) system.

The following report presents an overview of the waste management options considered by COM DEV.

2.2 Objectives of the Project

The following list outlines the major objectives of COM DEV's new waste treatment system.

i) Meet Waterloo Region requirements for discharge to sewer (½ of previous allowable maximum).
ii) Minimize waste sludge produced.
iii) Recover metals from rinse streams and plating baths for reuse.
iv) Minimize space requirements for the waste treatment system.
v) Minimize hazardous material handling for improved health and safety considerations.
vi) Minimize plating bath/part contamination.
vii) Minimize liability associated with off-site waste disposal.

2.3 Reasons of EMR Project

All actions toward pollution prevention must be aimed at the minimization of toxicity and total quantity of materials discharged. There are three categories in the field as follows:

1) Source reduction
2) Recycling
3) Treatment and disposal

Most of the reduction techniques are inexpensive and widely received in the industry. COM DEV also tries to adapt the source reduction philosophy, however, reduction techniques alone cannot eliminate process wastes entirely. Source reduction always has to be accompanied by other waste minimization techniques.

Previous System

COM DEV has been using the following waste treatment system along with outside disposal services in order to meet government standards and good business standards. The established techniques involved are:

1) Precipitation of dissolved metal with hydroxide or sulphide
2) Neutralization (pH control) with acid and/or sodium hydroxide
3) Oxidation of cyanide compounds by hypochlorite

Metal hydroxide sludge requires further processing to remove water for cost reduction. Most of COM DEV's plating waste solutions contain more than one metal and therefore have no economic value. Treatment and disposal, as discussed in previous sections, are being pressed by public and government toward "pollution prevention".

Recovery of heavy metals, therefore, is a natural selection for the treatment of plating wastes. Among the many technologies available in this category, they can be characterized according to the general technology employed. The following section overviews the existing technologies in the metal recovery area.

Electrodialysis

Employs alternating layers of ion exchange membranes permeable to either anions or cations but not both. When current is applied across the membranes two streams of solution are produced: concentrated inorganic salts and relatively pure water. The concentrated stream is typically added back to the process solution and the "pure" stream used as rinse water. The method does not remove organic materials and as such has found application in the purification of certain types of baths.

Reverse Osmosis

The pure water component of a waste stream is forced through a semi-permeable membrane under high pressure. As with electrodialysis the concentrated stream produced is typically returned to the process tank and the pure water stream used for further rinsing. Since only low molecular weight molecules can pass through the membrane the technique will segregate both the organic and inorganic components. It is, however, not as effective as electrodialysis in the concentration of dissolved materials.

Ultrafiltration

Waste water is forced through a semi-permeable membrane with a pore size which prevents the passage of high molecular weight organics but allows the dissolved salts through. It is an effective technique for the removal of oily wastes and colloidal materials but cannot be used to concentrate a process bath containing dissolved solids.

Electrolytic Metal Recovery

Dissolved metals are removed by deposition in electroplating cells specially designed to handle low concentration solutions. Use varies from the simple removal of metal from the waste stream to the production of high purity sheet stock which can be sold or reused directly (ie. as anode material). The technique is applicable, in theory at least, to any solution containing a metal which can be electroplated.

Ion Exchange

This is a liquid/solid extraction method whereby metal ions are replaced in solution by sodium or hydrogen ions. The method is amenable to a wide variety of discrete and mixed waste streams although some compounds will cause performance problems. It is capable of removing contaminants to very low levels and, as such, is used most commonly in the production of high purity water.

Evaporation

Waste water is distilled to a concentration which will permit its return to the process bath. Use of the technique varies from simple open evaporators to sophisticated vacuum systems. Due to its relatively simple operation and broad applicability this technique is used in the concentration and recovery of a wide variety of waste streams.

2.3.1 Selection of EMR

COM DEV relies on the quality of its plated finishes to provide the continued high product performance necessary to maintain its position as a high tech industry leader. Plating solution purity is of primary importance and most are prepared using reagent grade chemicals and deionized water. Currently, the dragouts employed on the copper and silver cyanide baths are added back as the plating solution level drops.

This has the effect of returning contaminants and breakdown products to the bath; risking plating finish integrity and shortening the usable life of the solution. Due to the nature of the electroless nickel and zincate plating solutions the dragouts employed for them cannot be added back.

Waste treatment techniques such as ion exchange and reverse osmosis are considered inappropriate for COM DEV's needs because they produce concentrated solutions which are intended to be readded to the plating bath. Simple precipitation of the dragouts would eliminate the need to add back or ship solutions for disposal but the result would be loss of salvageable metal (particularly silver) and increased sludge handling and disposal costs. Electrolytic metal recovery (EMR) is therefore considered to be the most appropriate waste treatment technique in COM DEV's case. EMR facilitates sludge reduction in combination with recovery of metal for reuse and/or salvage.

2.4 Outline of the Proposal Made to the MOEE

The proposal made to the Ministry of the Environment and Energy was for COM DEV to install equipment for the recovery of silver, copper, nickel, zinc, rhodium and gold from dragout solution and spent plating baths utilizing EMR cells. The following points were also proposed to the MOEE.

- EMR cells will operate in conjunction with a conventional precipitation/neutralization loop to recover salvageable metals from dragouts and spent plating solutions and to reduce overall sludge generation.

- Detailed parameters of proposed system and projected costs of the recovery and non-recovery portions. The completed system cost of approximately $190,000 with $65,000 attributable to the recovery portion.

- The expected payback period for the capital cost of the recovery portion to be 4½ years.

- Although short term capital cost by EMR/precipitation combination is higher than the precipitation technique alone, long term advantages of COM DEV's proposal meet the current trends in environmental legislation and COM DEV's general business philosophy.

3.0 THE PROJECT

The EMR project started. COM DEV obtained various information on equipment, materials and services as well as technical aspects. The following sections detail all aspects of this EMR project including implementation, installation, technologies, financial and other important areas need to be addressed to this project.

3.1 Implementation

COM DEV EMR cells, RETEC 07, were supplied by Kontek Ecology Systems Inc. in Burlington, Ontario. This system was chosen because it is a compact, inexpensive and efficient heavy metal recovery system. It is basically a plating cell. It contains seven (7) high surface area reticulated cathodes onto which metal is plated and eight (8) dimensionary stable anodes. A schematic of EMR system is shown on Figure 3.1-1 along with the DI waste and cyanide waste treatment systems. Since the volume of COM DEV's spent baths and dragout solutions are small, this system with a circulation pump is efficient enough to cover our needs at present and in the foreseeable future at COM DEV.

3.2 Installation/Commissioning

The metal recovery system was delivered after the construction and site preparation in 1992, followed by the system installation and commissioning through the spring of 1993. Although the majority of the installation was completed by early 1993, equipment malfunctions including pump breakages and cell leaks repeatedly delayed the completion of the commissioning. Meanwhile, the operation of metal recovery for copper and silver started intermittently between troubles and repairs. Zinc and nickel cells, despite frequent cell leaks and trouble, have started working after pH adjustment and close monitoring of the system. Closer relations and quicker response between the supplier and COM DEV would have saved time to complete commissioning. The result, however, is satisfactory and there was no serious problem during installation/commissioning period.

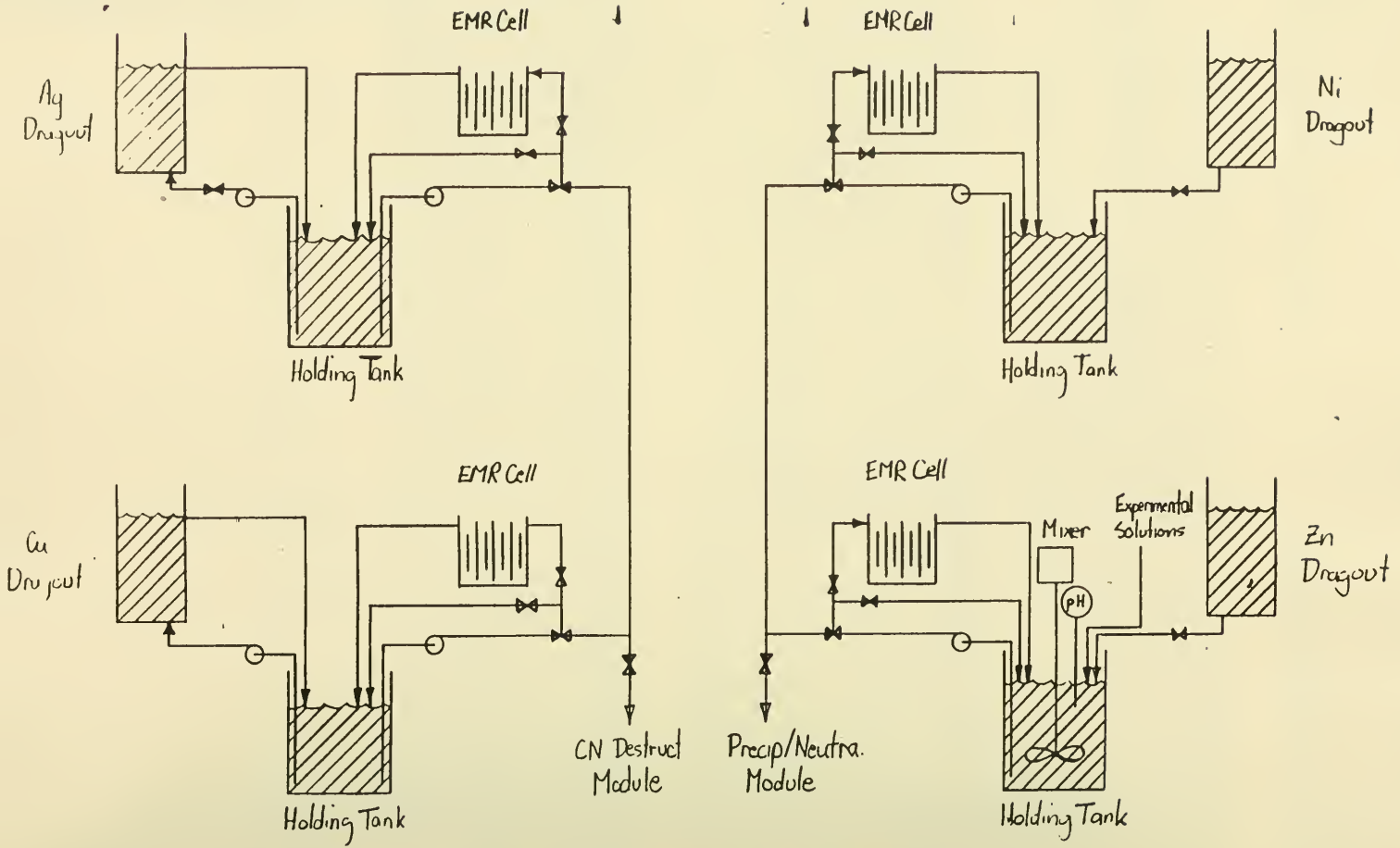

Figure 3.1-1: Schematic of EMR System

3.3 Technical

The recovery portion of the system consists of four electrolytic metal recovery (EMR) cells in a closed looped arrangement with four dragout tanks in the plating process. One of the cells is equipped with mixer and pH controls and is intended to be used for the R&D of EMR methods for other waste solutions. The EMR cells are integrated with the non-recovery portion of the system. This portion consists of a holding tank for waste dumps, a cyanide destruction module and a precipitation/neutralization module. Copper and silver dragouts are to be continuously recirculated through the EMR plating operations, but the project being delayed, this aspect of operation has not been proven. Nickel, zinc dragouts are static and these are treated on a batch basis as planned. It has been proven by initial operation that the concentrated spent baths of all four metals are treatable in EMR cells. COM DEV's main objective to reduce sludge generation has been accomplished in a small scale to date. What COM DEV needs to prove is continuous treatment at high efficiency in coming years. It is an achievable goal based on what COM DEV has learnt in the last few months of EMR operations.

An advantage of the EMR system included a partial destruction of cyanide during metal removal. The results support this prediction with excellent efficiency, reduced chemical cost and contamination from cyanide destruction. The results are reported in Section 4.5.

Following removal of the metal, the solutions are transferred to the precipitation/neutralization module for further treatment. The concept is working with all four EMR cells except the nickel cell for which spent solution has not been reached the low metal concentration yet.

3.4 Financial

The equipments and services required to install the EMR system and supporting processes are reported in the Table 3.4-1. The recovery portion in the table should be mentioned in this section in order to prove COM DEV's purchase and payment of the system. The following records are to support the claim we have made to the MOEE, although, exact prices paid of each item may not agree with the table.

Table 3.4-1: Equipment Cost

Item	Component	Qty	Cost			
			Non-Recovery	Recovery	R & D	Total
1	Collection tanks (Cu/Ag/Ni/Zn)	4	-	1506	502	2008
2	Mixer (for Zn tank)	1	-	-	886	886
3	Recirculation Pumps	6	-	3457	647	4104
4	EMR Cells (Retec 07)	4	-	12774	4258	17032
5	Cyanide Treatment Module	1	6182	-	-	6182
6	Waste Holding System	1	3920	-	-	3920
7	Precip/Neut System	1	4832	-	-	4832
8	Chemical Reagent tanks	3	1262	-	361	1623
9	Metering Pumps	8	2040	-	680	2720
10	Control Panel - Instrumentation	1	5547	-	725	6272
11	Control Panel - Equipment	1	5458	567	113	6138
12	Dragout tanks (Ag/Cu/Zn)	2	-	2775	-	2775
13	Exhaust System	1	3900	1950	650	6500
14	Power Supplies	4	-	4185	1395	5580
15	AA Spectrometer (complete)	1	6137	4602	1534	12273
16	Engineering Hours	200	9600	4800	1600	16000
	Sub total		48878	36616	13351	98845
17	Civil	-	15000	7500	2500	25000
18	Mechanical	-	-	715	-	715
19	Electrical	-	2880	1440	480	4800
20	Plumbing	-	11700	5850	1950	19500
21	Project Management	-	1699	849	283	2831
22	Installation	500 hr	24000	12000	4000	40000
	Total		104157	64970	22564	191691

3.4.1 Material/Service and Company List

Power Supplies	Allan Crawford	$5,340.00
A.A. Spectrometer	Varian Canada	$24,000.00
Piping & Labour/Materials	Ron Haddaway	$5,042.70
Waste Treatment Equipments	Kontek Ecology	$41,724.54 $11,921.30 $ 5,960.65 $59,606.49
Plastic Tanks	Miller Plastics	$1,900.00
Civil/Mechanical/ Piping/Electrical/ Engineering/Drawing/ Project Management	William Roberts	$38,722.60

3.5 Project Success/Failure Estimation

COM DEV, as described in the previous section, has not reached the point where it can judge whether this EMR project is a success or a failure due to the delay of the project. Technological advantages of the EMR system, however, are obvious by results obtained to date. The system concentrates all different metals from highly concentrated waste solutions. Results obtained for waste water effluent concentrations of silver, nickel, copper and zinc went less than a few ppm as was claimed. In this regard, this project is a success. Total elimination of the sludge waste may not be achieved, but the minimization is obvious and reasonable.

It appears the EMR system can handle the total waste that COM DEV produces based on the amount of plating COM DEV performs.

The cost analysis including operating cost, chemical cost, electrical cost, replacement cost can only be available when the EMR system be applied in the larger and continuing scale. The total estimated cost of the operation is:

Salvage value	$2,600/year decrease
Chemical costs	$3,625/year decrease
Disposal costs	$7,565/year decrease
Electricity	$672.00/year increase
Parts/Maintenance	$480.00/year increase
Labour	no change
Total Cost Difference	$12,638/year decrease

Based on what COM DEV has experienced, parts and maintenance costs may increase slightly depending on the stress level of the system. Electricity cost depends on whether COM DEV can find optimum recovery conditions on a continuous basis. Labour cost may be reduced once the condition has established. The actual waste treatment capacity looks the same as estimated. The key to the plating capacity depends on the metal plating efficiency at the very low (metal content of <30 ppm) level before the waste solution is transferred to cyanide oxidation and/or to the neutralization process.

4.0 RESULTS AND CONCLUSIONS

All four EMR cells have been working to extract metals from waste solutions. Metal concentration from each waste solution has been analyzed by Atomic Absorption Spectrophotometer. The summary of the results are described in the following section.

4.1 Zinc Recovery from Zincate

The zinc plating solution contains zinc metal and unknown chemicals that belong to supplier's trade secret. A fresh zincate plating tank contains approximately 1.1 g/l or 1100 ppm of zinc metal. A concentration of zinc will be reduced by the continuous extraction and by dilution when it is mixed with dragout solution. Figure 4.1-1 is the changing zinc concentration in the 250 l zinc waste tank. The vertical line represent zinc concentration against time (hrs) of EMR operation at electric current 15~20 A range. The solution pH has been maintained between 6 to 8 as suggested from zincate plating during these experiments. It is observed that the pH value tends to move towards alkaline as zinc concentration decreases and also it should be mentioned that extensive foaming (caused by additives of the solution) occurs at lower pH, which cannot be ignored as this foam contaminates the cell during plating.

4.2 Nickel Recovery

Nickel waste is a mixture of three nickel plating baths and each of the plating baths has a different chemical composition with different optimum pH ranges.

Metal recovery from nickel waste, we found, is sensitive to pH value and the following Figure 4.2-1 shows the total metal plating speed at different pH values. pH control was maintained by the addition of Ammonium hydroxide (NH_4OH) as the original pH of the nickel waste was low at 4.5. Ammonium hydroxide also produces complex compounds with nickel in alkaline condition and avoids precipitation (nickel hydroxide) that occurs when pH adjustment is carried out by NaOH. It is clear from this Figure 4.2-1 that the EMR Ni plating speed increases as pH increases and it reaches maximum around pH 8.0~8.5.

However, Cu metal deposition speed decreases dramatically as pH increases. Thus, pH control at 7.0 produces the best of the Ni and Cu. It is recommended to identify the concentration of Ni, Cu and other metals in the EMR system to have the most efficient use of the EMR cell.

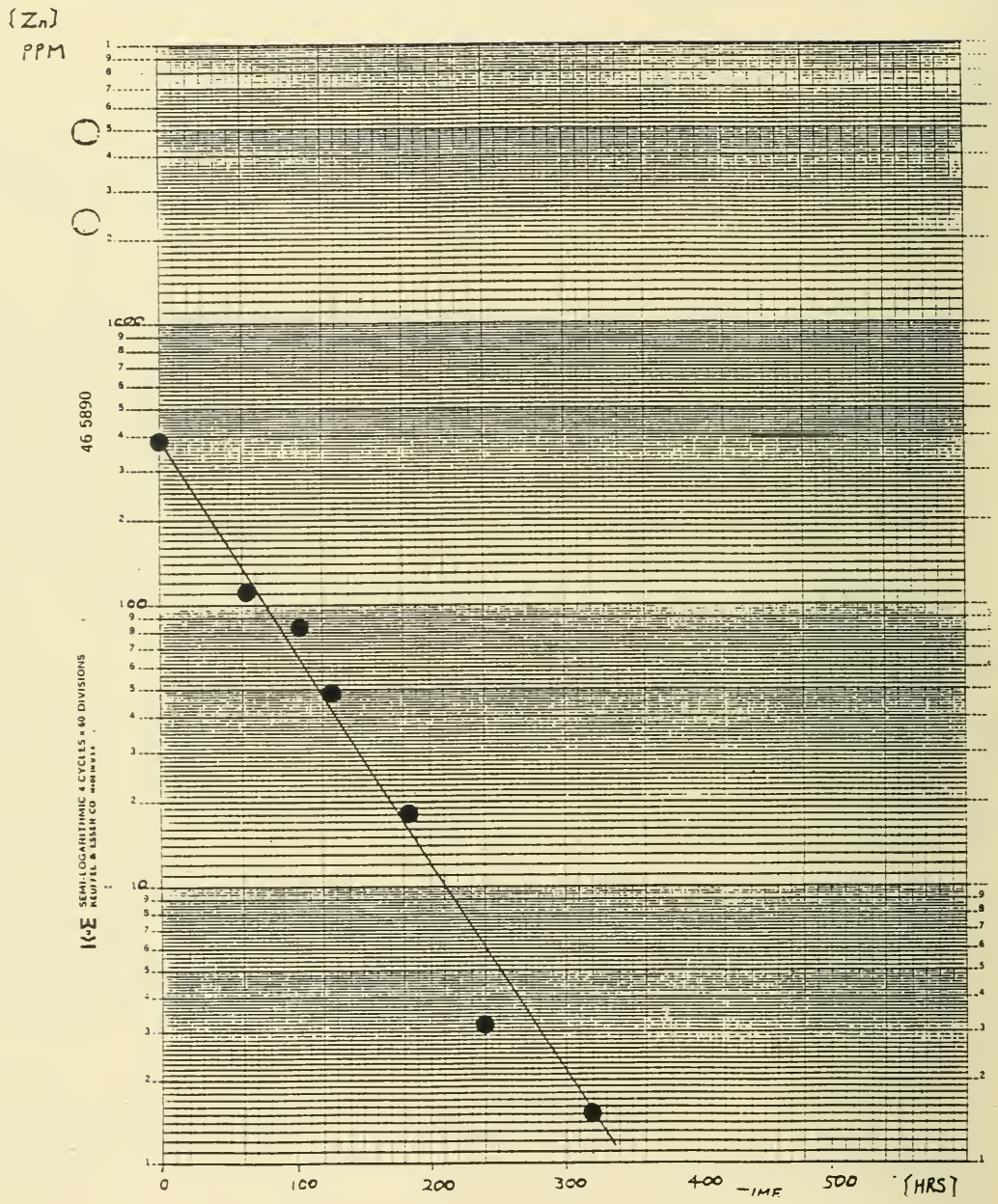

Figure 4.1-1: Zn Concentration ppm with Treating Time at 15–20 A

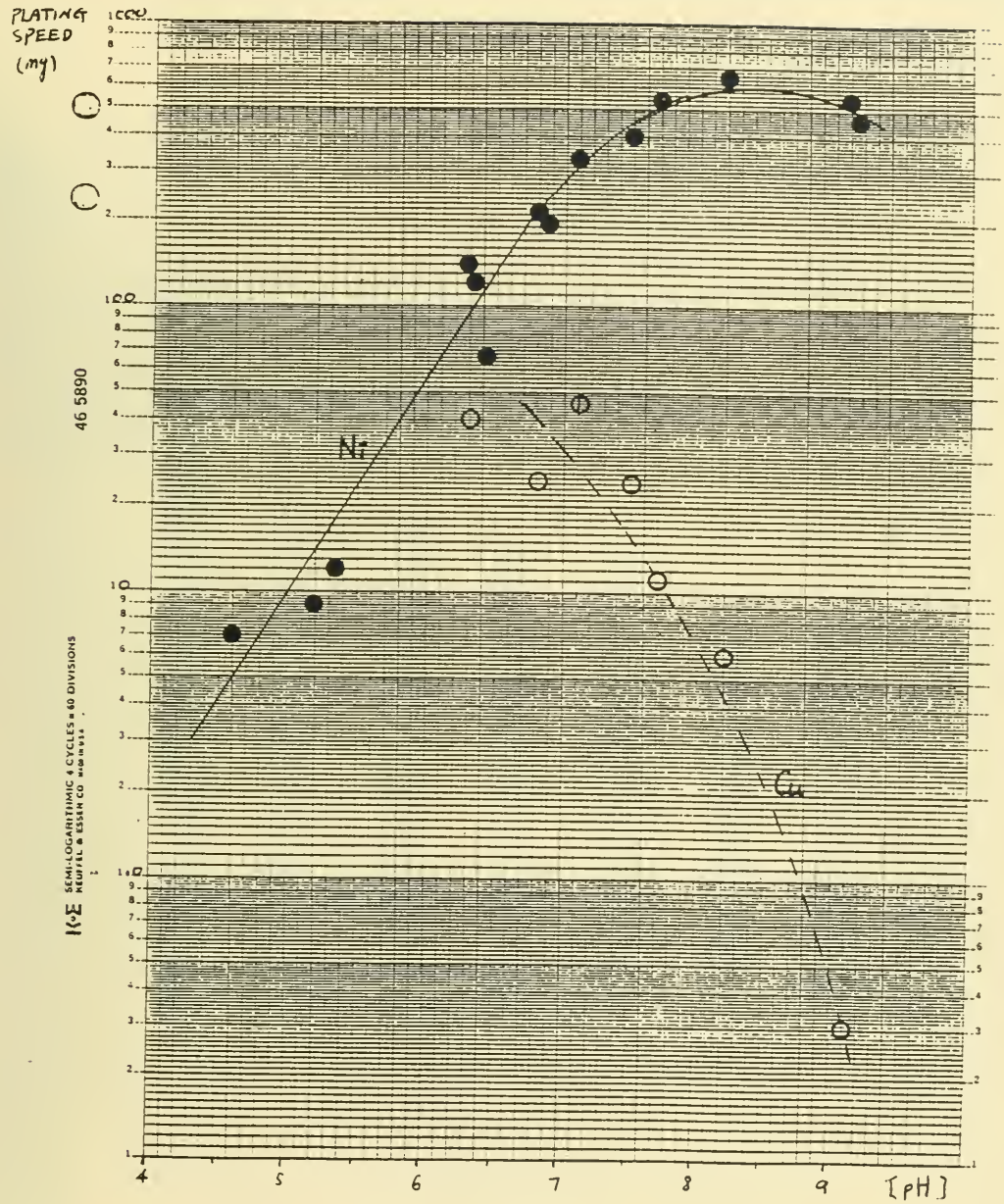

Figure 4.2-1: Total Metal Plating Speed for Nickel at Different pH Values
- 100cm^2 at 0.9 A at room temperature

4.3 Copper Recovery

The Cu waste solution contains cyanide compounds and therefore the EMR cell has to be operated at alkaline pH (pH>10.5). The cyanide bath being very efficient in plating, Cu recovery has been relatively easy and successful. Plated Cu metal in the EMR cell has been collected and the weight of all seven cathodes in the cell has been measured. The total Cu weight plated on the seven cathodes is 3254g and the Cu plated on each cathode is shown in Figure 4.3-1. As the Figure shows, the first cathode in the cell collects more Cu than the last cathode of the same cell which has been collecting only 70% of the first. It is understood that the first cell contacts with the highest concentration of the circulating waste and the collection efficiency decreases as Cu metal concentration decreases gradually toward the exit of the cell. This trend is confirmed in all other cells, although the gradient of the trend differs from cell to cell.

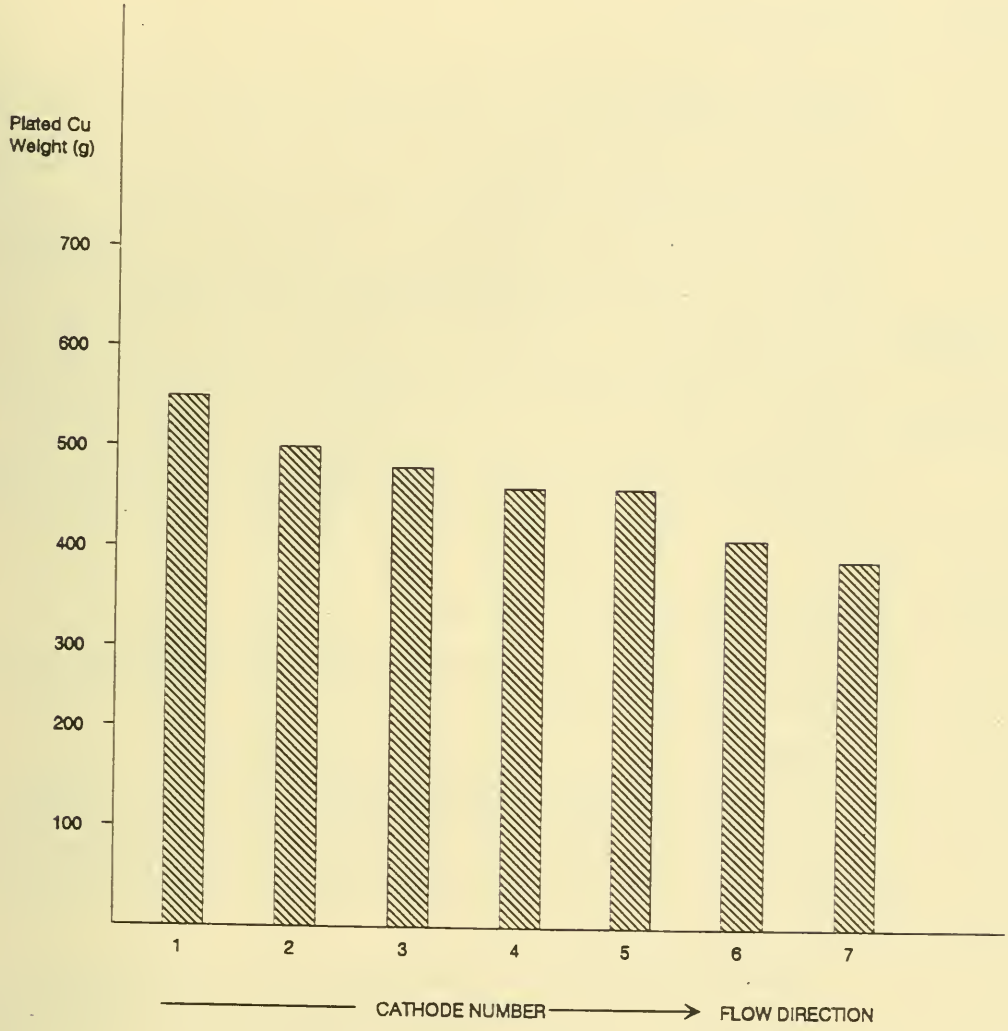

Figure 4.3-1: Weight of Plated Cu on Each Seven Cathodes
(Waste chemical flows through cathode 1 to 7 in a cell)

4.4 Silver Recovery

Silver EMR is similar to Cu system. The waste solution contains cyanide and it is operated at alkaline pH (pH>11.5). Metal deposition is relatively easy when the metal concentration is reasonably high, however, it becomes difficult to reach down to 5 ppm, the maximum Ag concentration allowed in waste water for sewage system. The EMR system collected 5890g of Ag metal to date and the Ag is to be shipped out to metal refinery. The unstable Ag concentration at low metal level, analyzed by the atomic absorption system, could be attributed to the fact that Ag plating is sensitive to current density and it tends to form weakly bonded metal grains on cathodes. These grains, if agitated, separate from these cathodes and dissolve into the chemicals once again, thus increasing total Ag concentration level. The other reason is that the Ag dissolves back into the cyanide based solution at this extremely low level to maintain chemical equilibrium. Analytical results during this fluctuating Ag level is shown in Figure 4.4-1. Solving this problem, it is recommended to keep EMR at very low current density and to plate Ag continuously without stopping electric current through the cathodes. COM DEV has achieved less than 3 ppm of Ag concentration by applying the above method.

4.5 Comparison of Objectives vs Results

Major objectives outlined in the introduction section are compared with the results obtained by EMR operations.

i) Meet Waterloo Region Requirements (½ of maximum limit)
Zn and Cu concentrations are easily achievable to 1.5 ppm (½ of 3.0 ppm). Ag concentration of 2.8 ppm has been achieved (½ of 5.0 ppm), but handling EMR cell at this level has to be careful. Plating Ni has not reached to this level (½ of 3.0 ppm limit) yet. Further experiment and process optimization may be required.

ii) Minimize Waste Sludge Produced
Total sludge produced with and without EMR system can be summarized in the following Table 4.5-1. The sludge is calculated from the form of metal hydroxides and metal sulfides as these are the final products when waste chemicals are sent outside for treatment.

The total reduction in sludge generation is 2019.8 kg (based on 5% net weight in water) or 109.9 kg of net solid metal salts.

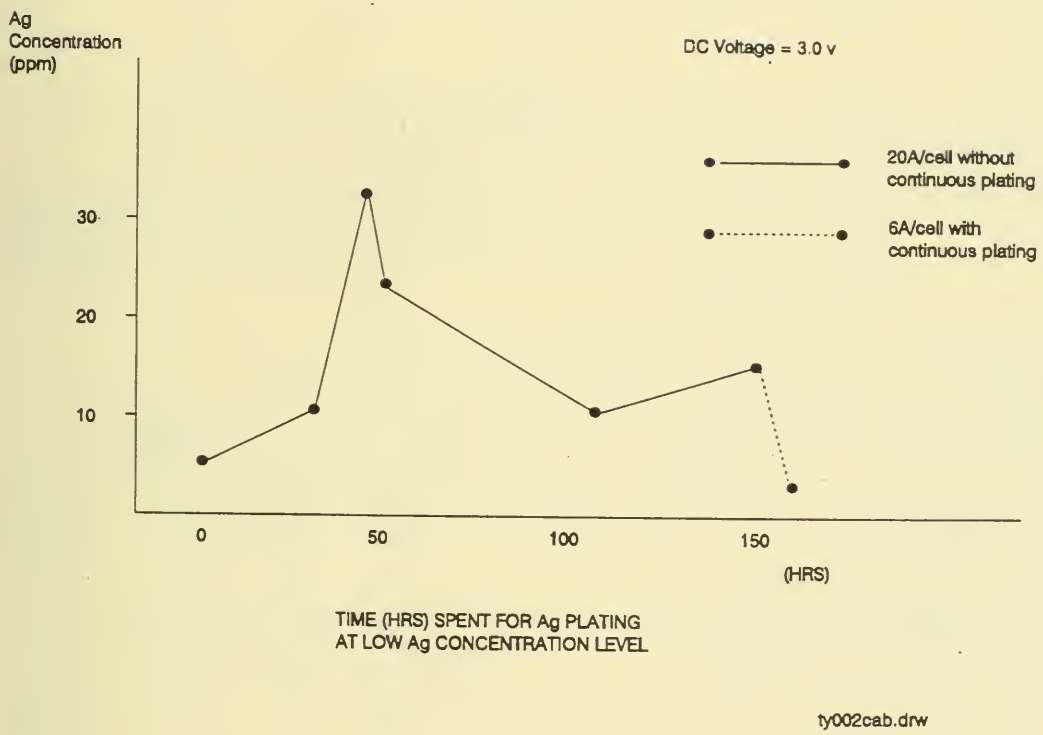

Figure 4.4-1: Ag Concentration Change in Waste Chemicals

Table 4.5-1: Estimated Total Sludge Produced Per Year (kg)

	Annual Quantity (kg)	
	With EMR	**Without EMR**
Zn	1.0	5.4
Ni	1.1	11.2
Cu	1.0	55.3
Ag	1.0	42.1

NOTE: Sludge weight is calculated as metal hydroxides and sulfides without including moisture in sludge. Ni and Zn to change to hydroxides, Cu and Ag to sulfides (mainly). These compounds are processed to make cement followed by permanent storage at secure disposal site.

Table 4.7-1: Do's and Don'ts

Actions	**Metal**			
	Zn	**Ni**	**Cu**	**Ag**
1. pH adjustment	YES	YES	YES	YES
2. Monitor metal contents	YES	YES	YES	YES
3. Electric current stop at low metal level	YES	YES	NO	NO
4. Mix with same metal wastes	NO	NO	NO	NO
5. Mix with same metal wastes (different baths)	YES	YES	YES	YES
6. High current density	YES	YES	YES	NO
7. High solution flow circulation	YES	YES	YES	YES - High (Ag) NO - Low (Ag)
8. Isolation of EMR cell from other cells	YES	YES	YES	YES
9. Re-use of plated metal for regular plating	NO	NO	NO	NO

iii) Metal Recovery for Reuse

All EMR cells are collecting metals at very high recovery efficiency as all metal concentration levels go down to a few ppm (except for Ni as it has not been experienced to this level). However, re-using the metals on-site has not been accomplished as these metals are recovered as a mixture and thus, need to be refined off-site.

iv) Minimize Space Requirements

The new EMR system is compact and efficient in using floor space. A 2' x 7' table is capable of holding all EMR cells and power supply for the cells. Since four 60 gallon holding tanks are placed underground, this system saved space and met the objective of the original plan.

v) Minimize Hazardous Material Handling

Since COM DEV does not need additional chemicals necessary to make metal precipitation, chemicals to destroy remaining cyanide and to neutralize before discharge are the only requirements for this system. Both sodium hypochlorite for cyanide and sodium hydroxide/hydrochloric acid are easy to handle. We have experienced even lower than standard cyanide concentration 10 to 12% of original value in both Cu and Ag solutions after these metals reached less than 3.0 ppm.

vi) Minimize Plating Bath/Part Contamination

The EMR cell does not produce a concentrate which can be directly added back to the process solution. There is therefore no concern regarding the accumulation of impurities or main bath components.

vii) Cost Savings

The cost analysis based on limited experience indicates that COM DEV can save a total of $12,638 per year as predicted in the beginning of the project. This reduction represents a pay back period on the capital cost of the recovery of the system of 4½ years.

viii) Minimize Liability

EMR cells have proven that they are capable of reducing heavy metals to less than the allowed maximum limit. COM DEV's capability to monitor these metals in used baths/dragouts minimizes possibilities of metal contaminations as long as the solutions are treated.

4.6 Changes

The original intention of the EMR system was to minimize sludge production and to maximize cleanliness, health safety and economical benefits. To date, the original plan has not been changed. However, due to COM DEV's business activities, this project has been delayed to obtain the full experimental data for full scale operation. Cyanide based Cu and Ag dragouts have not been recirculated through EMR system. All four EMR cells are, although they are proven working, yet to be operated at the best efficiency which include treatment speed, safety and economical aspects.

4.7 Summary of Do's and Don'ts in EMR System

The following Table 4.7-1 summarizes the Do's and Don'ts observed within limited EMR cell experience at COM DEV. All actions are simplified YES or NO for each metal.